CREEP OF HOT-PRESSED SiC-Al MATERIALS

R. MOUSSA, J.L. CHERMANT and F. OSTERSTOCK

Equipe Matériaux-Microstructure du L.A. 251, ISMra, Université, F-14032 Caen Cedex, France

Résumé - Deux nuances de SiC-Al pressées à chaud (avec 0.3% et 1.5% d'additifs) ont été déformées en flexion trois points dans le domaine de températures 1300°C - 1750°C. Elles ont la même taille de grains, mais les observations fractographiques montrent que les joints de grains sont de nature et de résistance différentes. L'effet résultant est un tracé d'Arrhénius courbe, pour l'une des deux nuances. La forme particulière du tracé d'Arrhénius est expliquée en considérant une contribution mécanique et athermique à la cavitation. Les mécanismes de fluage de base sont soit de la diffusion, soit de la dissolution-reprécipitation.

Abstract - Two batches of hot-pressed SiC-Al materials (0.3% and 1.5% additives) have been deformed in three points bending in the temperature range 1300°C - 1750°C. They have the same grain sizes, but fractographic investigations show that the grain boundaries are of different nature and strength. The resulting effect is a curved Arrhenius plot for one of the grades. The special shape of the Arrhenius plot is explained taking a mechanical and athermal contribution to the cavitation process into account. The basic creep mechanism is shown to be either diffusion or dissolution-reprecipitation.

I - INTRODUCTION

Among the thermomechanical ceramics, those based on SiC are very promising because of their good resistance to oxidation. This property is however limited by the open porosity of the sintered material which allows the corrosive species to penetrate into the bulk. Thus reaching high densities has been a target for the sintering development. Hot-pressing SiC with Al-based additives is one of the processes which has been developed and densities near the theoretical value are achieved.

However these materials contain a second amorphous phase between the grains and have thus a greater sensitivity to cavitation than SiC with B-based additives. These cavities will both provide paths for the corrosive species and give rise to rupture nuclei. Furthermore, if the cavitation acts during the creep test, the measured parameters will be influenced. Some phenomenological models have already been proposed for that phenomenon /1/2/.

The aim of this work is to investigate the creep behaviour of two batches of SiC-Al materials within this scope. On the scale of classical metallography, they have the same morphology. However fractographic investigations have shown that the grain boundary strength are different for the two batches, due to the different amounts of Al-additives used for the hot-pressing process /3/.

II - MATERIALS AND EXPERIMENTAL PROCEDURES

The two grades have been hot-pressed and furnished by the Elektroschmelzwerk Company (Kempten, F.R.G.). Amounts of 1.5% and 0.3% Al based additives have been used.

They will be denoted respectively as SiC-Al5 and SiC-AO3. In both grades, the grains are equiaxed and have a mean size of 1 μm . After grinding, chamferring and polishing, they have been deformed in the temperature range 1300°C - 1760°C under vacuum ($\sim 10^{-6}$ torr) in a Sesame-type furnace with tantalum heating elements. Further details can be found in references /3/ and /4/. Observations on fractured and tensile surfaces of deformed bending samples have been made using a JEOL T 20S scanning electron microscope.

III - RESULTS

The room and high temperature fracture behaviour of these materials had already been investigated during previous work /3/. It had been shown that the nature of the rupture (intergranular or transgranular) is strongly dependent of the amount of additives. The rupture of SiC-Al5 is intergranular from room temperature to high temperatures (1300°C). For materials containing only 0.75% of additives the rupture becomes mainly transgranular over the same temperature range. Observations on SiC-AO3 show also transgranular rupture even after high temperature deformation. Since these materials have all the same morphology, the evolution of the fractographic features may be interpreted as a change of the grain boundary strength. In the case of the SiC-Al materials, 0.7% additives seems to be a limit; it may be linked to the results of Y. Tajima and W.D. Kingery /5/ who showed that an amount

Fig. 1 - Fracture surface of SiC-AO3 grade after deformation at 1150°C.

of 0.7% Al can be dissolved in the SiC-crystals. It appears thus that the composition of the grain boundaries changes. Figures 1 and 2 show fracture patterns of the two materials used.

The creep measurements have been run in three-point bending. To calculate the creep rates, the simple transformation of the deflection rate, within the deviation from linearity assumption, has been used. The total deflection has been kept small ($< 1\text{mm}$). Thus the values of the stress exponent, n , and the apparent activation energy will not be much affected by the plastic singularity.

The results are summarized on figure 3 as Arrhenius plots ($\log \dot{\epsilon}$ vs $1/T$) where $\dot{\epsilon}$ is the value of the steady state creep rate and T the absolute temperature. Curves have been plotted for various values of the applied stress. Material SiC-AO3 exhibits a constant value of the apparent activation energy. It is a function of both temperature and applied stress.

Fig. 2 - SEM fractograph of SiC-Al5 grade showing the intergranular rupture and the flat facets at room temperature.

Fig. 3 - Arrhenius plots, for various applied stresses, for :

SiC-A03 grade,

SiC-Al5 grade.

Such a behaviour had been predicted by A. Venkateswaran and D.P.H. Hasselman /1//2/ after an analysis of the influence of cracks and cavities on the creep rates. Their analysis is based on the fact that firstly the presence of cracks enhances local stresses and thus the creep rate, and secondly that the latter is further enhanced by the increase of compliance due to the presence of cracks. The number of cracks influences only the creep rate in that it is shifted toward higher values whereas the apparent activation energy is influenced by that of crack growth during the creep process. The overall effect will be influenced mainly by the relative amounts of basic creep and the contribution of cavitation.

Concerning the basic creep mechanisms, SiC-Al₅ samples deformed at 1500°C have been thinned for TEM purposes. Results are given in figures 4 and 5. Figure 4 gives an overall view of several crystals : almost no dislocations can be found. This is confirmed by figure 5 which is a more enlarged view. The majority of dislocations are those coming from the stacking faults (the crystals are either 4H or 6H polytypes)/6/. Only few other types of dislocations can be seen, but they are not numerous enough or too localised to contribute to the deformation with an appreciable amount. Furthermore they may arise from powder milling. These results disagree with those obtained by C.H. Carter et al. /7/ on other types of SiC-materials (CVD, RBSC). Thus the basic creep mechanism may be either diffusion as already proposed by G. Grathwohl /8/ et al. or even dissolution-precipitation /9/, in accordance with the low values of the stress exponent, n ($1 < n < 2$).

Fig. 4 - Overall TEM view of a thin foil from a SiC-Al₅ sample deformed at 1500°C.

Fig. 5 - Isolated dislocations in a SiC-crystal after deformation at 1500°C.

The influence of cavitation on the apparent activation energy is governed by the activation energy of cavity growth. This is a process which is not accurately known and would need much work on the composition of the intergranular phase. However one may notice that the cavities are very elongated, with almost no separation of the crystals and that the decrease of the activation energy with decreasing temperature ($Q_A \rightarrow 0$) is a function of applied stress. Since the facets of the SiC-crystal are

very flat on the scale of the SEM, the mechanism proposed by A.G. Evans/10/ describing viscous cavity nucleation followed by mechanical rupture along the grain boundary may be applied here. Mechanical rupture is athermal and thus it contributes by zero to the activation and explains the shape of Arrhenius plots we have obtained.

IV - CONCLUSION

Taking into account the specific nature of the grain boundaries of two SiC-Al grades, we have been able to explain the special shape of the Arrhenius plot we have obtained with one of them. Mechanical rupture of the grain boundaries at low temperatures is favoured by the liquid-phase sintering and resulting flat facets as well by the residual amount of second phase remaining between the grains.

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